

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

| | | |
|--|-----------|---|
| (51) International Patent Classification ⁶: C07H 15/00, 17/00 | A1 | (11) International Publication Number: WO 96/15138 (43) International Publication Date: 23 May 1996 (23.05.96) |
| (21) International Application Number: PCT/US95/13999 (22) International Filing Date: 8 November 1995 (08.11.95) (30) Priority Data: 08/338,701 10 November 1994 (10.11.94) US 08/551,657 1 November 1995 (01.11.95) US (71) Applicant: HENKEL CORPORATION [US/US]; Suite 150, 140 Germantown Pike, Plymouth Meeting, PA 19462 (US). (72) Inventors: DESAI, Sureshchandra, G.; 123 Chopin Drive, Wayne, NJ 07470 (US). HESSEL, John, Frederick; 20 Clarendon Court, Metuchen, NJ 08840 (US). URFER, Allen, D.; 5697 North Brandywine Road, Shelbyville, IN 46176 (US). ALLEN, Charles, B.; 167 Percy Court, Norristown, PA 19401 (US). FISCHER, Stephen, A.; 307 Anderson Road, Yardley, PA 19067 (US). (74) Agent: DRACH, John, E.; Henkel Corporation, Suite 150, 140 Germantown Pike, Plymouth Meeting, PA 19462 (US). | | (81) Designated States: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LT, LU, LV, MD, MG, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TT, UA, UG, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG), ARIPO patent (KE, LS, MW, SD, SZ, UG). Published <i>With international search report.</i> |
| (54) Title: IMPROVED ALKYL POLYGLYCOSIDES (57) Abstract A composition and process for enhancing the aesthetic and tactile properties of an alkyl polyglycoside involving combining an alkyl polyglycoside having the general formula (I) RO(Z) _a , wherein R is a monovalent organic radical having from about 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6, with an aliphatic alcohol having from 1 to about 20 carbon atoms. | | |

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

| | | | | | |
|----|--------------------------|----|---------------------------------------|----|--------------------------|
| AT | Austria | GB | United Kingdom | MR | Mauritania |
| AU | Australia | GE | Georgia | MW | Malawi |
| BB | Barbados | GN | Guinea | NE | Niger |
| BE | Belgium | GR | Greece | NL | Netherlands |
| BF | Burkina Faso | HU | Hungary | NO | Norway |
| BG | Bulgaria | IE | Ireland | NZ | New Zealand |
| BJ | Benin | IT | Italy | PL | Poland |
| BR | Brazil | JP | Japan | PT | Portugal |
| BY | Belarus | KE | Kenya | RO | Romania |
| CA | Canada | KG | Kyrgyzstan | RU | Russian Federation |
| CF | Central African Republic | KP | Democratic People's Republic of Korea | SD | Sudan |
| CG | Congo | KR | Republic of Korea | SE | Sweden |
| CH | Switzerland | KZ | Kazakhstan | SI | Slovenia |
| CI | Côte d'Ivoire | LJ | Liechtenstein | SK | Slovakia |
| CM | Cameroon | LK | Sri Lanka | SN | Senegal |
| CN | China | LU | Luxembourg | TD | Chad |
| CS | Czechoslovakia | LV | Latvia | TG | Togo |
| CZ | Czech Republic | MC | Monaco | TJ | Tajikistan |
| DE | Germany | MD | Republic of Moldova | TT | Trinidad and Tobago |
| DK | Denmark | MG | Madagascar | UA | Ukraine |
| ES | Spain | ML | Mali | US | United States of America |
| FI | Finland | MN | Mongolia | UZ | Uzbekistan |
| FR | France | | | VN | Viet Nam |
| GA | Gabon | | | | |

IMPROVED ALKYL POLYGLYCOSIDES

Cross-Reference to related Applications

This application is a continuation-in-part of application serial number 08/338,701, filed on November 10, 1994, the entire contents of which are incorporated herein by reference.

Field of the Invention

The present invention relates to a composition and process for enhancing the tactile and aesthetic properties of alkyl polyglycosides. More particularly, by adding an effective amount of an additive to an alkyl polyglycoside, both the tactile and aesthetic properties of alkyl polyglycosides are significantly enhanced.

Background of the Invention

The aesthetic properties of alkyl polyglycoside solutions are apt to erode over a short period of time due to the crystallization of the alkyl polyglycoside. One

the alkyl polyglycoside solutions crystallize, the viscosity of the solutions increases to the point where handling problems result due to the non-homogeneity of the product. The crystallization phenomenon in fats and lipids is well known. All fats and lipids commonly form lamella-type structures, but every substance reveals different unit cell structures, i.e., polymorphism. The crystal structure of each modification depends on the geometrical conformation and chemical bonding of a molecule in which molecular interactions differ between different segments, such as an aliphatic chain, an olefin group in unsaturated fatty species, a methyl end group which stabilizes the lamella-lamella interface, a glycerol group in acylglycerol, -COOH in fatty acid, a polar head group in polar lipids, etc. Strongly hydrated nonionic surfactants have phase diagrams resembling those of ionic surfactants, suggesting the presence of a strong repulsive force between the micelles. These strong hydration repulsion forces are oftentimes balanced by van der Waals attractive forces between the lipid bilayers which provide for a uniform mixture.

It is well known that at a temperature below the critical transition temperature, a surfactant-water mixture exists in the so-called coagel and gel states, where the hydrocarbon chains of surfactant molecules are in a trans zigzag elongated state. A difference between the coagel and gel phases can be clearly recognized by the naked eye. That is, the gel phase is in a homogeneous,

semi-transparent state, while the coagel phases consist of a hydrated-crystalline state separated from the water solution phase. This is considered to be due to the difference in the mode of binding forces operating in the polar head region in the two phases. Probably, the predominant binding force in the coagel phase is electrostatic interaction between cationic head groups and their counter-ions, while there are in the gel phase hydration interactions of both with water molecules.

Physical deterioration of fat products such as margarine, shortening, and chocolate, just to name a few, depends on the size, morphology and polymorphic structure of the fat crystals, all of which are primarily influenced by the crystallization conditions, and secondly, by the phase transformation.

Kinetic parameters in the crystallization processes involve molecular clustering, solvation/desolvation, adsorption, surface/volume diffusion, conformational rearrangements and so on.

Crystallization involves two elementary processes: nucleation and crystal growth. Nucleation occurs when the solution or melt phases deviate from thermodynamic equilibria to a greater extent. Three actual nucleation phenomena occur in real systems. They are homogeneous nucleation which occurs in a very pure system and at a higher driving force. Heterogeneous nucleation predominates either in an impure system or at a lower driving force. Secondary nucleation which becomes

important when the pre-existing crystals provide secondary nuclei so that the precipitation is remarkably enhanced.

Certain alkyl polyglycosides have a tendency to crystallize during storage at ambient temperatures due to
5 their supersaturated state. Upon crystallization, the alkyl polyglycosides become turbid, i.e., cloudy and opaque in appearance due to the solids contained therein falling out of their supersaturated state which, when coupled with the resultant non-homogeneous state of the crystallized
10 solution and the attendant difficulties in handling operations such as pumping, is a highly undesirable phenomenon. In order to rectify this undesirable physical state, the alkyl polyglycoside composition must be heated in order to resolvate the crystals. As can be imagined,
15 this process is both expensive and time-consuming.

Another problem associated with alkyl polyglycosides relates to the undesirable tactile properties which they impart upon contact with the human body, commonly referred to in the industry as "scroopiness". More particularly,
20 alkyl polyglycosides, when applied onto hair and/or skin tend to make it feel rough, dry and sticky as if hairspray were applied thereon. In general, alkyl polyglycosides, due to their nonionic character, synergistic relationship with other surfactants, tendency towards high foaming and
25 mildness with respect to skin irritation, have become highly desirable surfactants for use in the personal care products industry. However, due to their tendency to impart this highly undesirable scroopiness effect when in

contact with the human body, their use in the personal care industry, in significant amounts, has been limited.

Typical commonly used hydrotropes such as ethanol and SXS have proven to be ineffective for maintaining the homogeneity of alkyl polyglycoside solutions, and thus inhibiting crystallization.

Consequently, it is the primary object of this invention to provide a composition and process of enhancing the aesthetic properties of alkyl polyglycosides by either reducing or eliminating their tendency to crystallize during prolonged storage under ambient conditions.

It is also an object of this invention to eliminate scroopiness in human hair and/or skin as a result of treatment with cleansing compositions containing alkyl polyglycosides.

Summary of the Invention

Other than in the operating examples, or where otherwise indicated, all numbers expressing quantities of ingredients or reaction conditions used herein are to be understood as modified in all instances by the term "about".

Briefly stated, the present invention is directed to a composition comprising a first alkyl polyglycoside having the general formula I

RO(Z)_n (I)

wherein R is a monovalent organic radical having from about 12 to about 16 carbon atoms; Z is a saccharide residue

having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6 and an additive selected from the group consisting of C_6 - C_{10} alkyl sulfates, unsaturated aliphatic carboxylic acids including their hydroxy-substituted derivatives or their salts, unsaturated aliphatic sorbitan esters, C_8 - C_{36} branched aliphatic di-carboxylic acids, C_6 - C_{34} branched aliphatic tri-carboxylic acids, alkyl sulfosuccinates, a second alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms, alkyl alkoxyates, alkyl and aryl phosphate esters, branched aliphatic carboxylic acids, unsaturated alcohols, Guerbet alcohols, alkoxyated C6 to C18 aliphatic polyglycosides, alkoxyated penterythritol, alkoxyated penterythritol esters, alkyl and aryl sulfonates, alkyl sulfonates, alkenyl sulfonates, alkyl amino carboxylates or imino dicarboxylates, betaines, carboxylated imidazoline derivatives, carboxylate surfactants, and mixtures thereof, in a weight ratio of first alkyl polyglycoside to additive of from about 500:1 to about 15:1, respectively.

The present invention also provides a process for enhancing the aesthetic and tactile properties of alkyl polyglycosides comprising adding an effective amount of an additive selected from the group consisting of C_6 - C_{10} alkyl sulfates, unsaturated aliphatic carboxylic acids including hydroxy-substituted derivatives thereof or their salts, unsaturated aliphatic sorbitan esters, C_8 - C_{36} branched aliphatic di-carboxylic acids, C_6 - C_{34} branched aliphatic tri-carboxylic acids, alkyl sulfosuccinates, a second alkyl

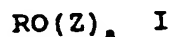
polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms, alkyl alkoxylates, alkyl and aryl phosphate esters, branched aliphatic carboxylic acids, unsaturated alcohols, Guerbet alcohols, alkoxylated C 6 to C 18 alkyl polyglycosides, alkoxylated penterithritol, alkoxylated penterithritol esters, alkyl and aryl sulfonates, alkyl sulfonates, alkenyl sulfonates, alkyl amino carboxylates or imino dicarboxylates, betaines, carboxylated imidazoline derivatives, carboxylates, and mixtures thereof, to a first alkyl polyglycoside of the formula I



wherein R is a monovalent organic radical having from about 12 to about 16 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6.

The present invention also provides a process for enhancing the tactile properties of alkyl polyglycoside solutions when applied onto human hair and/or skin by contacting human hair and/or skin with a composition comprised of:

(a) an alkyl polyglycoside of formula I:



wherein R is a monovalent organic radical having from about 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; a is a number having a value from 1 to about 6 and an effective amount of a dicarboxylic acid having from 8 to about 36 carbon atoms;

(b) a salt of an alkyl ether sulfate having an average

d gree of ethoxylation of about 2; and

(c) an N-alkoxylated fatty acid amide, thereby imparting to said human hair, skin and combinations thereof a feeling which is free of tackiness, roughness, dryness
5 and combinations thereof.

Brief Description of the Drawings

Fig. 1 is a bar graph illustrating the effect on viscosity at a temperature of about 72°F, when various unsaturated fatty acids such as EMERSOL® 315, an
10 unsaturated carboxylic acid, EMPOL® 1008, an unsaturated aliphatic dicarboxylic acid, and EMSORB® 2500, an unsaturated alkyl sorbitan ester, are added at a 1.5% actives level to GLUCOPON® 625 CS SURFACTANT at about 40% actives.

15 Fig. 2 is a bar graph illustrating the effect on viscosity, at a temperature of about 72°F when the unsaturated fatty acids of Figure 1 are added at a 1.5% actives to GLUCOPON® 625 CSUP SURFACTANT at about 40% actives.

20 Fig. 3 is a bar graph comparing the initial viscosity at 72°F when the unsaturated fatty acids of Figure 1 are added at a 1.5% actives to GLUCOPON® 625 CSUP SURFACTANT at about 40% actives versus the viscosity after approximately six weeks of storage at about 45°C.

25 Fig. 4 is a bar graph comparing the initial viscosity at 72°F when the unsaturated fatty acids of Figure 1 are added at a 1.5% actives to GLUCOPON® 625 CS SURFACTANT at about 40% actives versus the viscosity after approximately

six weeks of storage at about 45°C.

Fig. 5 is a bar graph illustrating the effect on the amount of foam formed at 72°F in deionized water when the unsaturated fatty acids of Figure 1 are added at a 1.5%
5 actives to GLUCOPON® 625 CS SURFACTANT at about 40% actives, as measured by the Ross Miles test.

Fig. 6 is a bar graph illustrating the effect on the amount of foam formed at 72°F in deionized water when the unsaturated fatty acids of Figure 1 are added at a 1.5%
10 actives to GLUCOPON® 625 CSUP SURFACTANT at about 40% actives, as measured by the Ross Miles test.

Fig. 7 is a bar graph illustrating the effect on wetting ability when the unsaturated fatty acids of Figure 1 are added at a 1.5% actives to GLUCOPON® 625 CS
15 SURFACTANT at about 40% actives at a temperature of about 72°F in deionized water, as measured by the Draves Wetting test.

Fig. 8 is a bar graph illustrating the effect on wetting ability when the unsaturated fatty acids of Figure 1 are added at a 1.5% actives to GLUCOPON® 625 CSUP
20 SURFACTANT at about 40% actives at a temperature of about 72°F in deionized water, as measured by the Draves Wetting test.

Detailed Description of the Invention

25 In general, it is desirable to improve both the

aesthetic and tactile properties of alkyl polyglycosides.

An alkyl polyglycoside is a sugar derivative surfactant which is typically less irritating to human skin than other surfactants. Also, though it is a nonionic surfactant, an alkyl polyglycoside forms a stable foam per se, and furthermore, exerts a foam-stabilizing effect when combined with anionic surfactants. Conventional body cleansers such as shampoos, whether for hair or body, contain anionic surfactants as their major components.

Because of the low skin irritation associated with the use of alkyl polyglycosides as the nonionic surfactant, the use of alkyl polyglycosides as an ingredient has become an option in the personal care industry. However, prior to their admixture into cleaning compositions, there is a time period during which the alkyl polyglycosides are shipped from the manufacturer and stored in drums. It is during this time period, which may be very short, and under ambient conditions, the alkyl polyglycosides have a tendency to crystallize, thus becoming turbid and more viscous. Moreover, as was mentioned above, the use of alkyl polyglycosides as hair and/or skin cleansers, until now, has been limited because of the previously-described undesirable scroopiness effect imparted by alkyl polyglycosides when in contact with the human body. Thus, these alkyl polyglycosides are used primarily in small quantities for their synergistic relationship with other surfactants, low skin irritation and tendency to high foaming.

It has now surprisingly been found that by adding an effective amount of the disclosed additives to alkyl polyglycosides, alkyl polyglycosides may be used as the primary surfactant in a personal care product formulation, while at the same time exhibiting significantly enhanced aesthetic properties based on the elimination or reduction in crystallization, along with the resultant increase in viscosity of the alkyl polyglycosides.

Thus, according to the present invention, there is provided a composition having improved aesthetic and tactile properties comprising a mixture of a first alkyl polyglycoside of formula I



wherein R is a monovalent organic radical having from about 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6, and an additive selected from the group consisting of C₆-C₁₀ alkyl sulfates, unsaturated aliphatic carboxylic acids including hydroxy-substituted derivatives thereof or their salts, unsaturated aliphatic sorbitan esters, C₈-C₃₆ branched aliphatic di-carboxylic acids, C₆-C₃₄ branched aliphatic tri-carboxylic acids, alkyl sulfosuccinates, a second alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms, alkyl alkoxyates, alkyl and aryl phosphate esters, branched aliphatic carboxylic acids, unsaturated alcohols, Guerbet alcohols, alkoxyated C 6 to C 18 alkyl

polyglycosides, alkoxylated penterythritol, alkoxylated penterythritol esters, alkyl and aryl sulfonates, alkyl sulfonates, alkenyl sulfonates, alkyl amino carboxylates, or imino dicarboxylates, betaines, carboxylated
5 immidazoline derivatives, carboxylate surfactants, and mixtures thereof.

In the aspect of the present invention which relates to the elimination of the crystallization of alkyl polyglycosides,
10 the preferred alkyl polyglycosides are those which are most susceptible to crystallization upon storage. Such alkyl polyglycosides are alkyl polyglucosides wherein the alkyl groups contain from 12 to 16 carbon atoms and the sugar residue is derived from glucose. Such alkyl polyglucosides
15 which are made by reacting a mixture of fatty alcohols having 12 to 16 carbon atoms and glucose and are available commercially, for example, from Henkel Corporation as GLUCOPON® 600 and GLUCOPON® 625 SURFACTANT, or APG® 600 and APG 625 SURFACTANT.

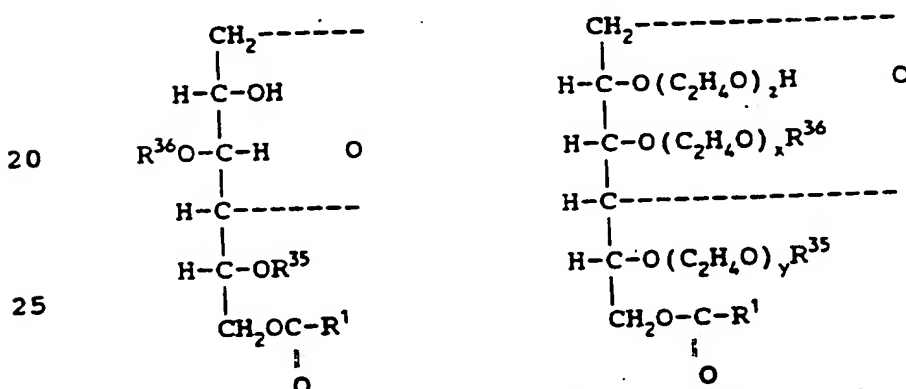
20 These alkyl polyglycosides have an average degree of polymerization, which is typically defined as the average number of repeat units, or in this case glucose units, per polymer chain, in the range of about 1.4 to about 1.6. The pH of the alkyl polyglycoside is typically in the range of
25 about 6 to about 12. The percent actives of the alkyl polyglycosides employed in the present invention is in the range of about 5 to about 80%, and preferably about 40%.

The C₆-C₁₀ alkyl sulfates which may be employed are

short-chain alkyl sulfates characterized by the formula $R'-SO_3^-X^+$ wherein X is Na, K, Mg, and NH_3 . These can be manufactured from alkyl alcohols by sulfation with SO_3 or chlorosulfonic acid.

5 The unsaturated aliphatic carboxylic acids, including their hydroxy-substituted derivatives and salts thereof, employed in the present invention have from 6 to 22 carbon atoms. Preferred unsaturated fatty acids include: linoleic acid and its sodium or potassium salt (commercially
10 available under the tradename EMERSOL® 315); linseed fatty acid (commercially available under the tradename EMERY 643); or hydroxy-substituted unsaturated aliphatic carboxylic acids such as ricinoleic acid, and mixtures thereof.

15 Unsaturated aliphatic sorbitan esters of the type



20 wherein each of R^{35} and R^{36} is independently hydrogen or OCR^1 and wherein R^1 is a C_{10-22} hydrocarbon radical with the proviso that $x+y+z$ is a number from 5-30, may also be used.
25

30 Dimer fatty acids and salts thereof having the structure $\text{X}-\text{R}^2-\text{X}$ where R^2 is a C_{8-36} hydrocarbon radical and X is COOH may also be employed. Examples include the
35 potassium salt of dimer acid (commercially available under

the tradename EMPOL 1008).

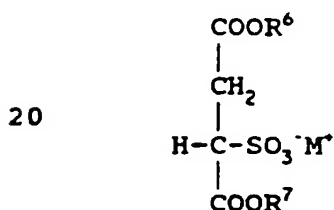
Similarly, trimer fatty acids having from about 6 to about 54 carbon atoms may also be employed as an additive.

Aliphatic dicarboxylic acids of the formula $R^3-CX-(COOH)_2$ wherein R^3 is a C_{16-18} hydrocarbon radical and X is NH_2 or $N(R^4)_2$, where R^4 is a C_{1-4} hydrocarbon radical may also be used.

Short-chain alkyl polyglycosides wherein the alkyl group has from about 4 to about 10 carbon atoms may also be used to inhibit crystallization.

Alkyl alkoxyates having the structure $R^5-(EO)_v-(PO)_w-OH$ wherein R^5 is a C_{8-22} hydrocarbon radical, v is a number from about 0 to about 100 and w is a number from about 0 to about 100, such that $v + w \geq 1$, may also be employed.

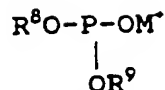
Yet another additive which may be employed are the alkyl sulfosuccinates characterized by the structure



wherein each of R^6 and R^7 is independently a C_{8-18} linear or branched, saturated or unsaturated hydrocarbon radical or H, with the proviso that when R^6 is H, R^6 does not equal R^7 , and M^+ is Na, K or ammonium.

Alkyl and aryl phosphate esters characterized by the structure



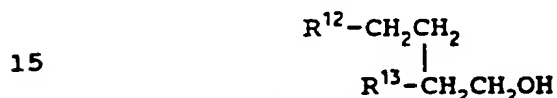


wherein each of R^8 and R^9 is independently a C_{8-22} straight or
 5 branched, saturated or unsaturated hydrocarbon radical
 which may be ethoxylated with from 0 to 20 moles of
 ethylene oxide, or H, with the proviso that when R^8 is H, R^8
 does not equal R^9 , and M^* is H, Na, K, or ammonium.

Branched fatty acids of the $\text{R}^{10}-\text{COOH}$ type where R^{10} is
 10 a C_{4-22} hydrocarbon radical.

Unsaturated alcohols of the $\text{R}^{11}-\text{OH}$ type may also be
 employed.

Guerbet alcohols having the formula

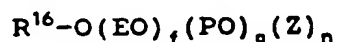


15 wherein each of R^{12} and R^{13} is independently a C_{8-10} straight
 or branched chain hydrocarbon radical, and also esters of
 Guerbet alcohols of the formula $\text{R}^{14}\text{COOR}^{15}$ where

20 R^{14} is $\text{R}^{12}-\text{CH}_2\text{CH}_2$

and R^{15} is a C_{8-18} hydrocarbon radical.

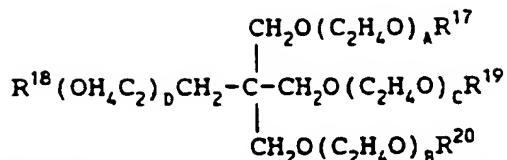
Another useful additive is an alkoxyated alkyl
 25 polyglycoside having the formula



where Z is a glucose residue and n is a number from about
 1 to 5, f is from about 0 to 100 and g is from about 0 to
 100 where $f + g \geq 1$.

30 Ethoxylated penterythritol and its esters having the
 formula

16



5

where $A+B+C+D$ is a number from 40-200 and wherein each of $\text{R}^{17}-\text{R}^{20}$ is independently H or OCR^{40} wherein R^{40} is a C_{6-22} hydrocarbon radical.

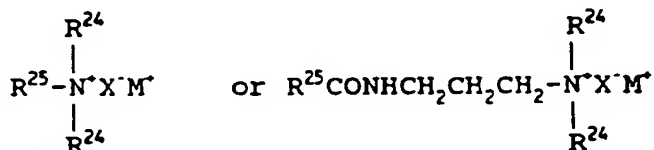
Aliphatic or alkyl and aryl sulfonates having the
10 formula

$\text{R}^{21}-\text{SO}_3^-\text{M}^+$ wherein R^{21} is a C_{8-22} straight or branched chain, saturated or unsaturated alkyl and aryl group, may also be employed.

Alkyl amino carboxylates or imino dicarboxylates
15 having the formula $\text{R}^{22}-\text{NR}^{23}\text{CH}_2\text{CH}_2\text{COO}^-\text{M}^+$ wherein R^{22} is a C_{8-18} aliphatic radical, linear or branched, saturated or unsaturated, R^{23} is H or $\text{CH}_2\text{CH}_2\text{COO}^-$ and M is Na, K or ammonium.

Betaines having the formula:

20



25

wherein R^{25} is a C_{8-22} straight or branched chain, saturated or unsaturated aliphatic radical, R^{24} is methyl or 2-hydroxy ethyl, X^- is CH_2COO^- or $\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{SO}_3^-$, and M^+ is Na, K or ammonium.

Carboxylated imidazoline derivatives of the type
30 $\text{R}^{26}\text{CONHCH}_2\text{CH}_2\text{NR}^{27}\text{R}^{28}$ wherein R^{26} is a C_{8-22} alkyl group, straight or branched chain, saturated or unsaturated, R^{27} is CH_2COONa or $\text{CH}_2\text{CH}(\text{OH})\text{CH}_2\text{SO}_3\text{Na}$, and R^{28} is $\text{CH}_2\text{CH}_2\text{OH}$ can also be employed.

Carboxylate surfactants of the formula $R^{29}-COOM^+$ wherein R^{29} is C_{8-22} alkyl group, straight or branched chain, or $R^{30}CONCH_2CH_2$ with R^{30} is a C_{8-22} group (i.e. sarcosinate), and M^+ is Na, K or ammonium.

5 The compositions according to the invention can also contain viscosity-controlling additives such as aliphatic alcohols having from 1 to about 20 carbon atoms, a particularly preferred aliphatic alcohol being ethanol. A preferred amount of viscosity-controlling agent is from
10 about 1% to about 20% by weight.

In a preferred embodiment of the present invention the weight ratio of first alkyl polyglycoside to additive, present in the composition, is from about 500:1 to about 15:1, respectively. A particularly preferred ratio is
15 about 25:1. The percent actives of the alkyl polyglycosides is in the range of from about 5-80%, and most preferably about 40%.

According to another aspect of the present invention, there is also provided a process for enhancing the
20 aesthetic and tactile properties of alkyl polyglycosides involving adding an effective amount of an additive to a first alkyl polyglycoside of formula I:



wherein R is a monovalent organic radical having from about
25 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6. The additives which may be

employed are the same as those described above.

In a preferred embodiment of the present invention, an alkyl polyglycoside according to formula I is heated to a temperature in the range of from about 35 to about 70°C, and preferably about 40°C. The additive is then added to the heated alkyl polyglycoside, with mixing, in a weight ratio of about 500:1 to about 15:1, and preferably about 25:1. The addition of these types of additives has been shown to be enhance the aesthetic and tactile properties of alkyl polyglycosides without adversely affecting their physical and performance properties.

The practice of this invention may be further appreciated by consideration of the following non-limiting, working examples, and the benefits of the invention may be further appreciated by reference to the comparison examples.

CRYSTALLIZATION INHIBITION:

EXAMPLES

Alkyl polyglycoside compositions listed in Table I were prepared. The crystallization inhibiting effect of each composition was evaluated according to the following method. The results are also shown in Table I.

Evaluation Method:

In Examples 1-4 and Comparative Example 1, 40 grams of 50% actives GlucoPON®625 CS SURFACTANT, an alkyl polyglycoside composition commercially available from

Henkel Corp., Ambler, PA was charged into a beaker and heated to a temperature of about 40°C. Various additives were then added to the alkyl polyglycoside composition with thorough mixing. The compositions were then allowed to stand under ambient conditions for a predetermined number of days. The Examples were then evaluated for appearance to determine whether the formation of crystals was visible to the human eye. The Examples were then tested to determine their viscosities, foaming, wetting, and critical micelle concentration, the results of which can be found in Figures 1-8.

TABLE I

| No. | EMERSOL® 315 (K) | EMERSOL® 315 (Na) | EMPOL® 1008 | EMSORB® 2500 | APPEARANCE |
|-----|---------------------|----------------------|----------------|-----------------|------------|
| 1 | 1.5 | -- | -- | -- | clear |
| 2 | -- | 1.5 | -- | -- | clear |
| 3 | -- | -- | 1.5 | -- | clear |
| 4 | -- | -- | -- | 1.5 | clear |
| C1 | -- | -- | -- | -- | hazy |

* (all components are represented in grams)

EMERSOL® 315 (K) is the potassium salt of linolic acid, commercially available from Henkel Corp., Emery Division, at 1.5% actives.

EMERSOL® 315 (Na) is the sodium salt of linolic acid, commercially available from Henkel Corp., Emery Division, at 1.5% actives.

EMPOL 1008 is the potassium salt of dimmer acid,

commercially available from Henkel Corp., Emery Division,
at 1.5% actives.

EMSORB® 2500 is sorbitan mono oleate, commercially
available from Henkel Corp., Emery Division, at 1.5%
5 actives.

Examples 5-8 and Comparative Example 2 listed in Table
II were prepared as per above using 80 grams of 40% actives
Glucopon®625 csup, an alkyl polyglycoside composition also
10 commercially available from Henkel Corp. The Examples were
then evaluated and tested, as per above.

TABLE II

| No. | EMERSOL ® 315 (K) | EMERSOL® 315 (Na) | EMPOL® 1008 | EMSORB® 2500 | APPEARANCE |
|-----|-------------------------|----------------------|----------------|-----------------|------------|
| 5 | 1.5 | -- | -- | -- | clear |
| 6 | -- | 1.5 | -- | -- | clear |
| 7 | -- | -- | 1.5 | -- | clear |
| 8 | -- | -- | -- | 1.5 | clear |
| C2 | -- | -- | -- | -- | hazy |

* (all components are represented in grams)

20 As can be seen from the results in Tables I and II, as
well as Figures 1-8, the addition of one of the disclosed
additives such as an unsaturated fatty acid having a
terminal anionic group thereon significantly inhibits or
delays the formation of crystals in alkyl polyglycosides
25 of formula I, without impairing its physical properties.

Thus enhancement of an alkyl polyglycoside's aesthetic appearance is desired, i.e., the inhibition of crystallization during storage at ambient conditions, the disclosed additive is preferably added in a weight ratio of 5 alkyl polyglycoside to additive of about 25:1.

TACTILE IMPROVEMENT:Composition A

336.0 grams of PLANTAREN®-2000 (an alkyl polyglycoside commercially available from Henkel Corp., Emery division, was heated to 50°C, after which 26.9 grams of EMPOL® 1008 was added. The pH of the solution was adjusted to 7.2 with 11.6 grams of 25% NaOH solution. 18.7 grams of water was then added to the solution to adjust the solids to about 50%. The solution was then cooled to 25°C. The final product was clear yellow in appearance having a pH of 7.2 and a Brookfield viscosity of about 2,950 cps.

The following shampoo formulations were prepared in order to determine the tactile properties imparted by an alkyl polyglycoside composition of the present invention versus that of a control group.

EXAMPLE 9

| | <u>COMPONENT</u> | <u>% by weight</u> |
|----|------------------------------|--------------------|
| | STANDAPOL® ES-2 | 15.0 |
| | STANDAMID® SD | 3.0 |
| 20 | Composition A | 15.0 |
| | thickener | 0.5 |
| | Citric acid soln., pH to 6.5 | 2.0 |
| | water | <u>64.5</u> |
| | | 100.0 |

STANDAPOL® ES-2 is ammonium laureth sulfate having a

degree of ethoxylation of about 2, available from Henkel Corp., Emery Division.

STANDAMID® SD is cocamide DEA, available from Henkel Corp., Emery Division.

5

COMPARISON EXAMPLE 3

| | <u>COMPONENT</u> | <u>% by weight</u> |
|----|------------------------------|--------------------|
| | STANDAPOL® ES-2 | 15.0 |
| | STANDAMID® SD | 3.0 |
| | PLANTAREN®-2000 | 15.0 |
| 10 | thickener | 0.5 |
| | Citric acid soln., pH to 6.5 | 2.0 |
| | water | <u>64.5</u> |
| | | 100.0 |

Test Procedure:

15 Example 9 and Comparison Example 3 were used to wash human hair and then evaluated for the feel they imparted on the hair after washing. The results showed that those individuals using the alkyl polyglycoside composition of the present invention found their hair to be soft and
20 supple after washing. Conversely, those washing with Comparison Example 3 found that their hair felt very dry and tacky immediately following the washing. Thus, it can be seen that by incorporating the alkyl polyglycoside composition of the present invention into personal care
25 products, the resultant tactile properties are significantly enhanced.

It should be noted, however, that in a process for

cleansing human hair and/or skin, the amount of additive will vary, depending on the particular type of cleansing formulation being employed. Typical hair shampoos contain the following components: C₈₋₂₂ alkyl sulfates and their salts which may be ethoxylated with from 1-50 moles of (EO), cocoamides their salts and derivatives thereof, along with citric acid, its salts and derivatives. Thus, the amount of additive to be added will to a certain extent depend on the additional components being employed, other than alkyl polyglycoside. However, where the components include: (a) a first alkyl polyglycoside in combination with an additive selected from the group consisting of C₆-C₁₀ alkyl sulfates, unsaturated aliphatic carboxylic acids including hydroxy-substituted derivatives thereof or their salts, unsaturated aliphatic sorbitan esters, C₈-C₃₆ branched aliphatic di-carboxylic acids, C₆-C₃₄ branched aliphatic tri-carboxylic acids, alkyl sulfosuccinates, a second alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms, alkyl alkoxylates, alkyl and aryl phosphate esters, branched aliphatic carboxylic acids, unsaturated alcohols, Guerbet alcohols, alkoxylated C 6 to C 18 alkyl polyglycosides, alkoxylated penterithritol, alkoxylated penterithritol esters, alkyl and aryl sulfonates, alkyl sulfonates, alkenyl sulfonates, alkyl amino carboxylates or imino dicarboxylates, betaines, carboxylated imidazoline derivatives, carboxylate surfactants, and mixtures thereof; (b) alkyl sulfates and their salts; (c) fatty acid amides, the percent actives ratio of components (a) + (b):(c) will

typically fall in the range of about 1:6 to about 1:4, and preferably about 1:1. The same holds true for body cleansers. The preferred additive for use in body cleanser formulations is a dicarboxylic acid having from about 8 to
5 about 36 carbon atoms.

It will be recognized by those skilled in the art that changes may be made to the above-described embodiments of the invention without departing from the broad inventive concepts thereof. It is understood, therefore, that this
10 invention is not limited to the particular embodiments disclosed, but is intended to cover all modifications which are within the scope and spirit of the invention as defined by the appended claims.

The invention claimed is:

1. An alkyl polyglycoside composition comprising:

(a) a first alkyl polyglycoside having the general formula I

5 $RO(Z)_a$ (I)

wherein R is a monovalent organic radical having from about 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6, and

10 (b) an additive selected from the group consisting of C_6 - C_{10} alkyl sulfates, unsaturated aliphatic carboxylic acids including hydroxy-substituted derivatives thereof or their salts, unsaturated aliphatic sorbitan esters, C_8 - C_{36} branched aliphatic di-carboxylic acids, C_6 - C_{54} branched aliphatic tri-
15 carboxylic acids, alkyl sulfosuccinates, a second alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms, alkyl alkoxyates, alkyl and aryl phosphate esters, branched aliphatic carboxylic acids, unsaturated alcohols, Guerbet alcohols, alkoxyated C 6 to C 18 alkyl
20 polyglycosides, alkoxyated penterythritol, alkoxyated penterythritol esters, alkyl and aryl sulfonates, alkyl sulfonates, alkenyl sulfonates, alkyl amino carboxylates or imino dicarboxylates, betaines, carboxylated imidazoline derivatives, carboxylate surfactants, and mixtures thereof,
25 in a weight ratio of alkyl polyglycoside to additive in the range from about 500:1 to about 15:1.

2. The composition of claim 1 wherein said ratio of said first alkyl polyglycoside to said additive is about 25:1.
3. The composition of claim 1 wherein said additive is an unsaturated aliphatic carboxylic acid including its hydroxy-substituted derivative or its salt.
4. The composition of claim 1 wherein said additive is an alkyl or alkenyl sulfonate.
5. The composition of claim 1 wherein said additive is an alkyl amino carboxylate or imino dicarboxylate.
- 10 6. The composition of claim 1 wherein said additive is a C₆-C₁₀ alkyl sulfate.
7. The composition of claim 1 wherein said additive is a C₈-C₃₆ branched aliphatic di-carboxylic acid.
8. The composition of claim 1 wherein said additive is a C₆-C₃₆ branched aliphatic tri-carboxylic acid.
- 15 9. The composition of claim 1 wherein said additive is an unsaturated aliphatic sorbitan ester.
10. The composition of claim 1 wherein said additive is an alkyl sulfosuccinate.
- 20 11. The composition of claim 1 wherein said additive is a second alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms.
12. The composition of claim 1 wherein said additive is an alkyl alkoxylate.
- 25 13. The composition of claim 1 wherein said additive is an alkyl and aryl phosphate ester.
14. The composition of claim 1 wherein said additive is a branched aliphatic carboxylic acid.

15. The composition of claim 1 wherein said additive is an unsaturated alcohol.
16. The composition of claim 1 wherein said additive is a Guerbet alcohol.
- 5 17. The composition of claim 1 wherein said additive is an alkoxyated C₈₋₁₈ alkyl polyglycoside.
18. The composition of claim 1 wherein said additive is an alkoxyated penterythritol or its ester.
- 10 19. The composition of claim 1 wherein said additive is an alkoxyated penterythritol ester.
20. The composition of claim 1 wherein said additive is an alkyl and aryl sulfonate.
21. The composition of claim 1 wherein said additive is a betaine.
- 15 22. The composition of claim 1 wherein said additive is a carboxylated imidazoline derivative.
23. The composition of claim 1 wherein said additive is a carboxylate surfactant.
24. The composition of claim 1 wherein said first alkyl
20 polyglycoside is an aqueous solution having a percent actives in the range from about 5 to about 80%.
25. The composition of claim 24 wherein percent actives is about 40%.
26. The composition of claim 1 having a pH of about 7.2.
- 25 27. The composition of claim 1 wherein said salt of said unsaturated aliphatic carboxylic acid is selected from the group consisting of potassium salt of linoleic acid, a sodium salt of linoleic acid, potassium salt of linolenic

acid, sodium salt of linolenic acid and mixtures thereof.

28. A process for enhancing the aesthetic and tactile properties of an alkyl polyglycoside comprising adding an effective amount of an additive selected from the group consisting of C₆-C₁₀ alkyl sulfates, unsaturated aliphatic carboxylic acids including hydroxy-substituted derivatives thereof or their salts, unsaturated aliphatic sorbitan esters, C₈-C₃₆ branched aliphatic di-carboxylic acids, C₆-C₃₆ branched aliphatic tri-carboxylic acids, alkyl sulfosuccinates, a short-chain alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms, alkyl alkoxylates, alkyl and aryl phosphate esters, branched aliphatic carboxylic acids, unsaturated alcohols, Guerbet alcohols, alkoxylated C 6 to C 18 alkyl polyglycosides, alkoxylated penterythritol, alkoxylated penterythritol esters, alkyl and aryl sulfonates, alkyl sulfonates, alkenyl sulfonates, alkyl amino carboxylates or imino dicarboxylates, betaines, carboxylated imidazoline derivatives, carboxylate surfactants, and mixtures thereof, to a first alkyl polyglycoside of formula I:



wherein R is a monovalent organic radical having from about 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; and a is a number having a value from about 1 to about 6.

29. The process of claim 28 wherein said first alkyl polyglycoside and additive are present in a weight ratio of from about 500:1 to about 15:1, respectively.

30. The process of claim 29 wherein said ratio is about 25:1.

31. The process of claim 28 wherein said additive is an unsaturated aliphatic carboxylic acid including its hydroxy-substituted derivative or its salt.

32. The process of claim 31 wherein said unsaturated aliphatic carboxylic acid is in a salt form.

33. The process of claim 32 wherein said salt of said unsaturated aliphatic carboxylic acid is selected from the group consisting of potassium salt of linoleic acid, a sodium salt of linoleic acid, potassium salt of lenolenic acid, sodium salt of lenolenic acid and mixtures thereof.

34. The process of claim 28 wherein the weight ratio of said first alkyl polyglycoside to said additive is about 25:1.

35. The process of claim 28 wherein said additive is an alkyl or alkenyl sulfonate.

36. The process of claim 28 wherein said additive is an alkyl amino carboxylate or imino dicarboxylate.

37. The process of claim 28 wherein said additive is a C₆-C₁₀ alkyl sulfate.

38. The process of claim 28 wherein said additive is a C₈-C₃₆ branched aliphatic di-carboxylic acid.

39. The process of claim 28 wherein said additive is a C₆-C₃₆ branched aliphatic tri-carboxylic acid.

40. The process of claim 28 wherein said additive is an unsaturated aliphatic sorbitan ester.

41. The process of claim 28 wherein said additive is an

alkyl sulfosuccinate.

42. The process of claim 28 wherein said additive is a short-chain alkyl polyglycoside wherein the alkyl group has from 4 to 10 carbon atoms.

5 43. The process of claim 28 wherein said additive is an alkyl alkoxylate.

44. The process of claim 28 wherein said additive is an alkyl and aryl phosphate ester.

10 45. The process of claim 28 wherein said additive is a branched aliphatic carboxylic acid.

46. The process of claim 28 wherein said additive is an unsaturated alcohol.

47. The process of claim 28 wherein said additive is a Guerbet alcohol.

15 48. The process of claim 28 wherein said additive is an alkoxylated C₈₋₁₈ alkyl polyglycoside.

49. The process of claim 28 wherein said additive is an alkoxylated penterythritol.

20 50. The process of claim 28 wherein said additive is an alkoxylated penterythritol ester.

51. The process of claim 28 wherein said additive is an alkyl and aryl sulfonate.

52. The process of claim 28 wherein said additive is a betaine.

25 53. The process of claim 28 wherein said additive is a carboxylated imidazoline derivative.

54. The process of claim 28 wherein said additive is a carboxylate surfactant.

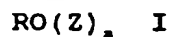
55. The process of claim 28 wherein said first alkyl polyglycoside is an aqueous solution having a percent actives in the range from about 5 to about 80%.

56. The process of claim 55 wherein said percent actives
5 is about 40%.

57. A process for cleansing human hair or skin comprising contacting human hair, skin and combinations thereof with a cleansing composition comprised of:

(a) an alkyl polyglycoside of formula I:

10



wherein R is a monovalent organic radical having from about 8 to about 18 carbon atoms; Z is a saccharide residue having 5 or 6 carbon atoms; a is a number having a value from 1 to about 6 and an effective amount of a dicarboxylic
15 acid additive having from about 8 to about 36 carbon atoms;

(b) a salt of an alkyl ether sulfate having an average degree of ethoxylation of about 2; and

(c) an N-alkoxylated fatty acid amide, thereby
20 imparting to said human hair, skin and combinations thereof a feeling which is free of tackiness, roughness, dryness and combinations thereof.

58. The process of claim 57 wherein said dicarboxylic acid is selected from the group consisting of azelaic acid, sebacic acid, dimer acid, and mixtures thereof.
25

59. The process of claim 58 wherein said dicarboxylic acid is a dimer acid having about 36 carbon atoms.

60. The process of claim 57 wherein R in formula I is an alkyl group having from about 12 to about 16 carbon atoms.

61. The process of claim 57 wherein a in formula I is a number from about 1.4 to about 1.6.

5 62. The process of claim 57 further comprising adding the salt of an alkyl ether sulfate having an average degree of ethoxylation of about 2 and an N-alkoxylated fatty acid amide.

63. The process of claim 57 wherein said salt of an alkyl
10 ether sulfate is sodium laureth sulfate having an average degree of ethoxylation of about 2.

64. The process of claim 57 wherein said N-alkoxylated fatty acid amide is cocamide DEA.

65. The process of claim 57 wherein components (a)-(c) are
15 combined in a percent actives ratio of (a)+(b):(c) of from about 1:6 to about 1:4.

66. The process of claim 65 wherein said ratio of (a)+(b):(c) is about 1:1.

66. The composition of claim 1 further comprising an
20 aliphatic alcohol having from 1 to about 20 carbon atoms.

67. The composition of claim 66 wherein said alcohol is ethanol.

68. The process of claim 28 wherein said additive is further comprised of an aliphatic alcohol having from 1 to
25 about 20 carbon atoms.

69. The process of claim 68 wherein said alcohol is ethanol.

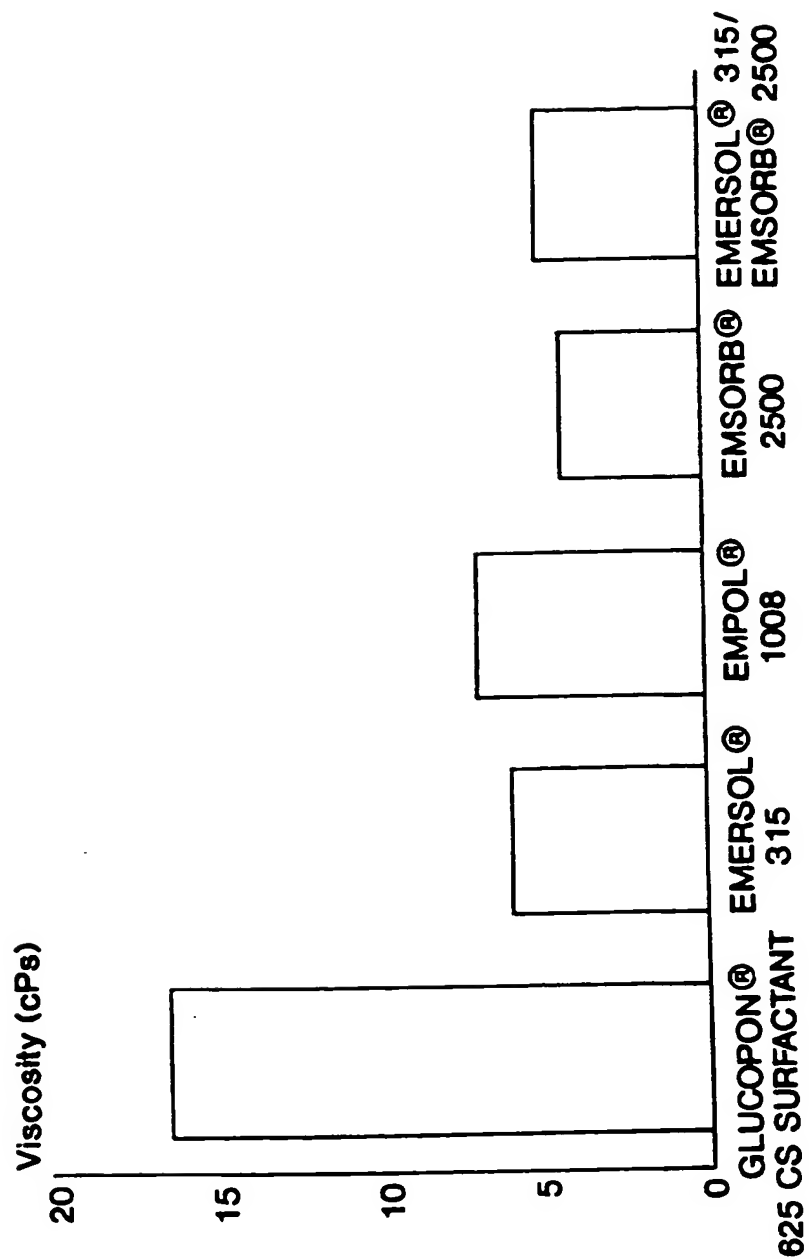


FIG. 1

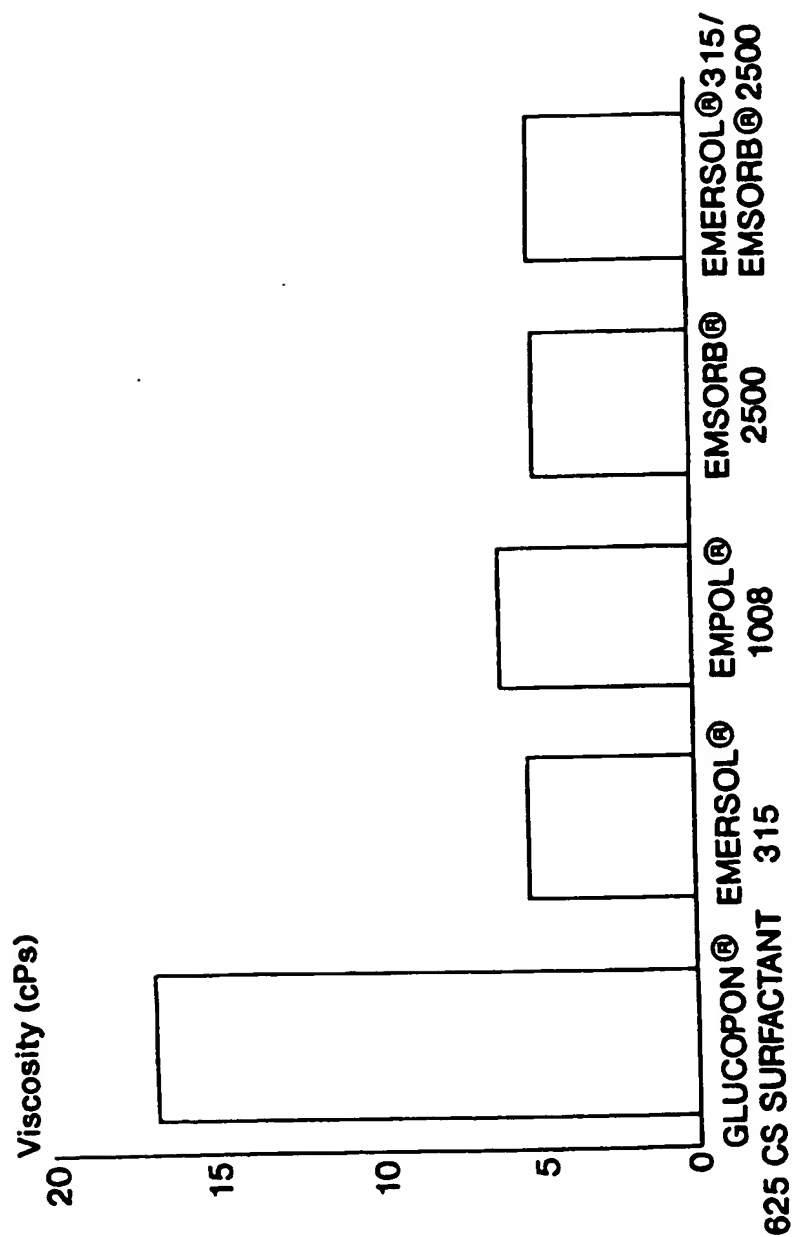


FIG. 2

3/8

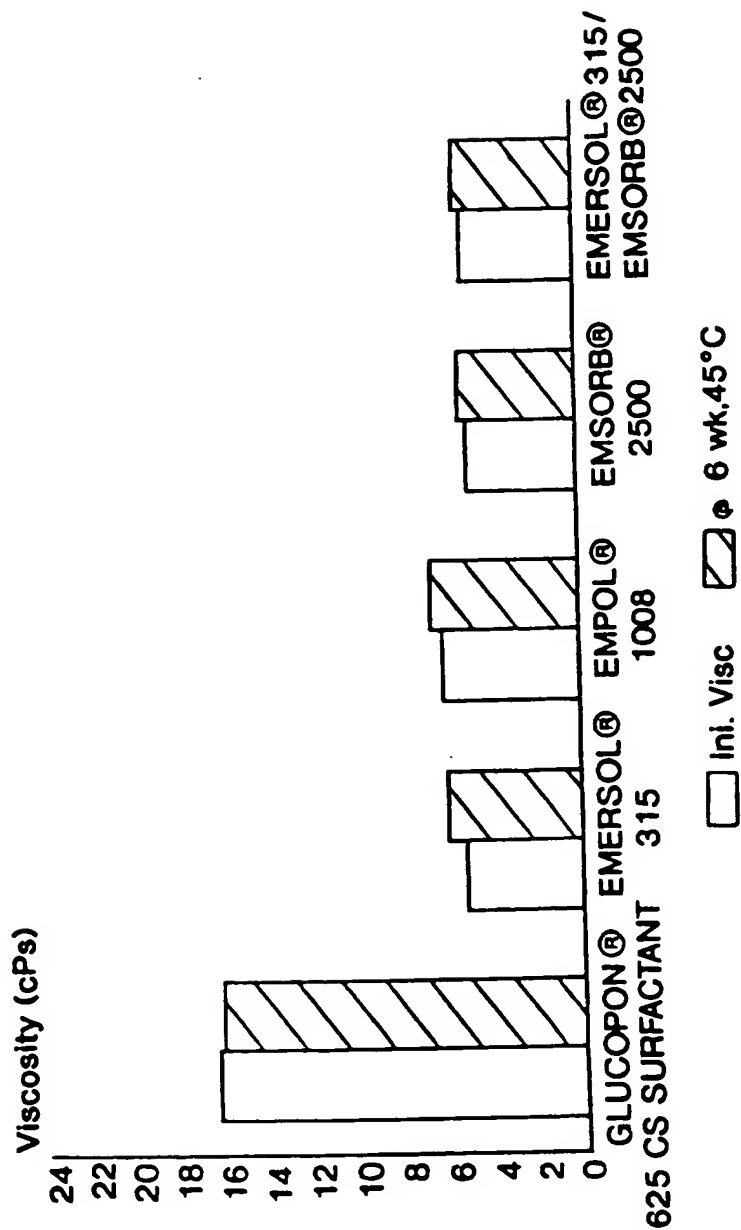


FIG. 3

4 / 8

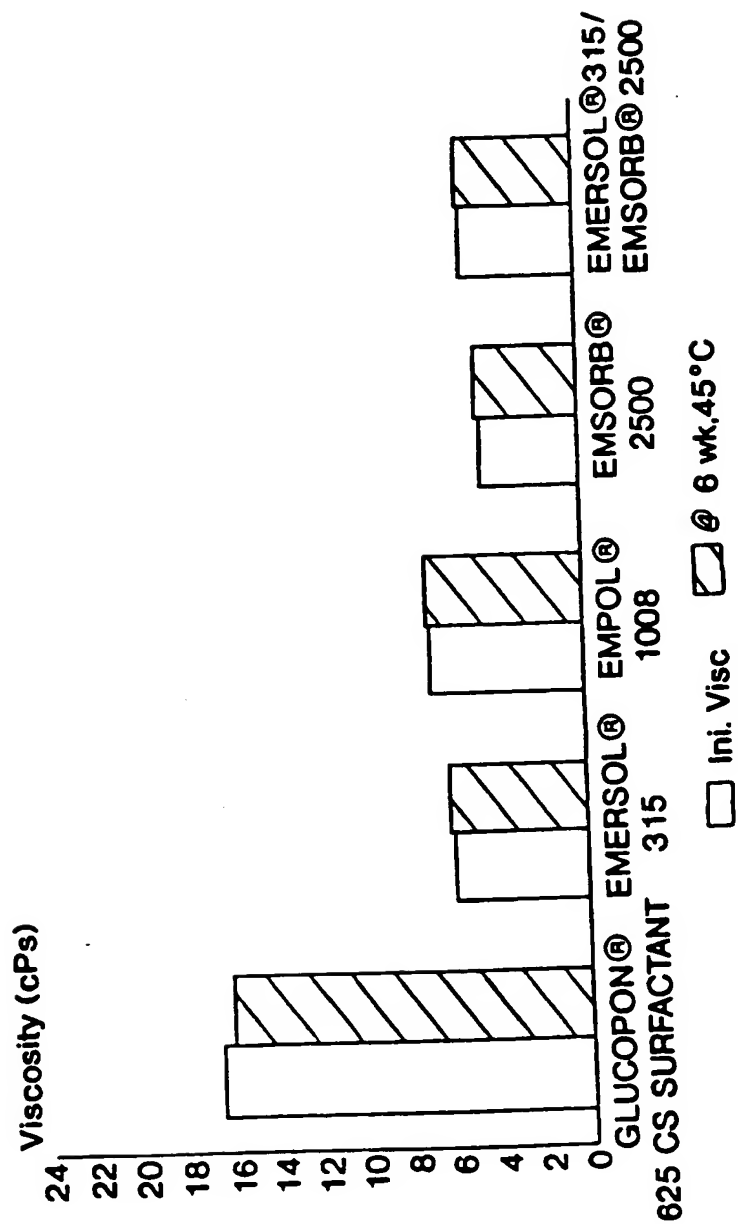


FIG. 4

SUBSTITUTE SHEET (RULE 26)

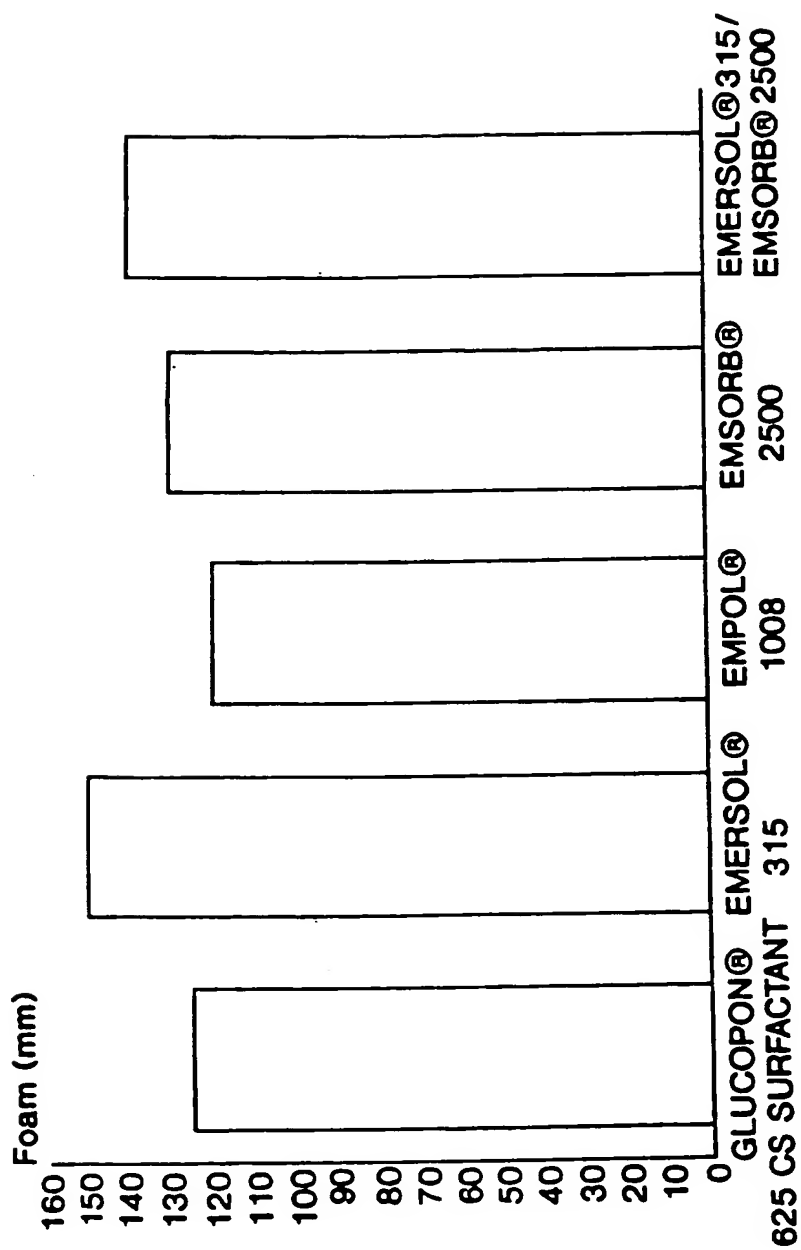


FIG. 5

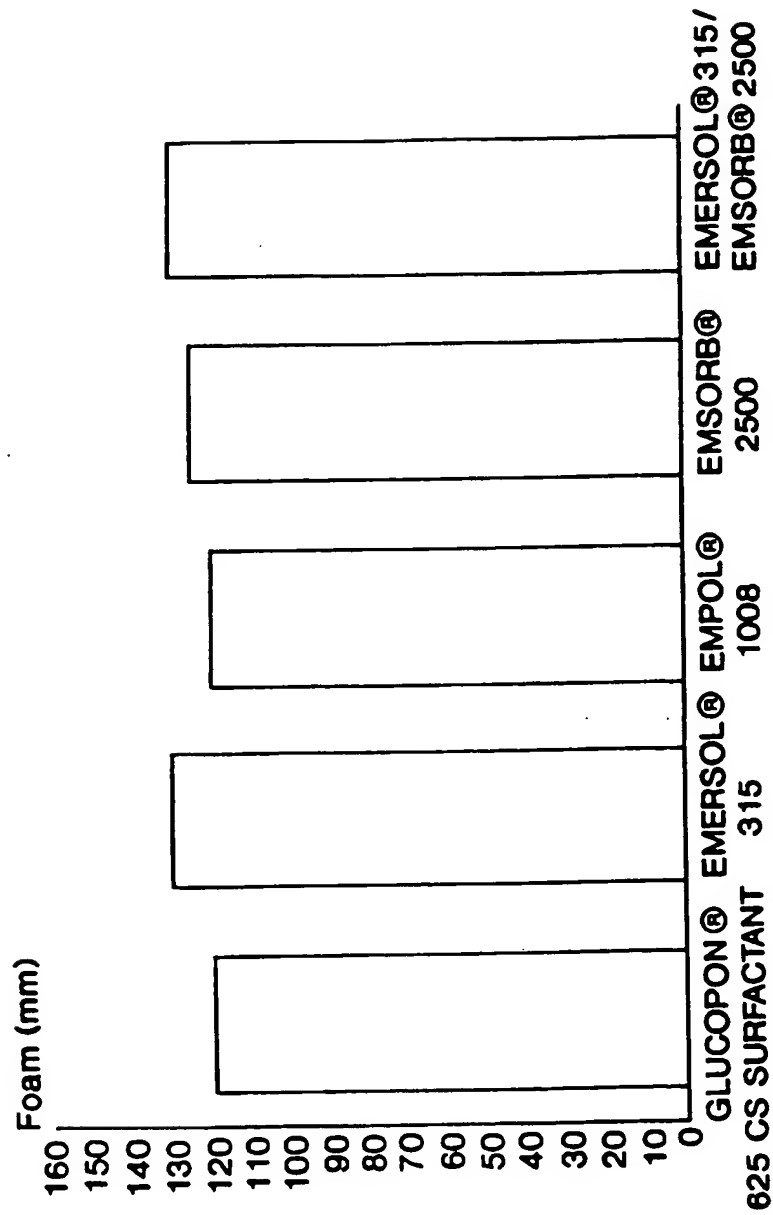


FIG. 6

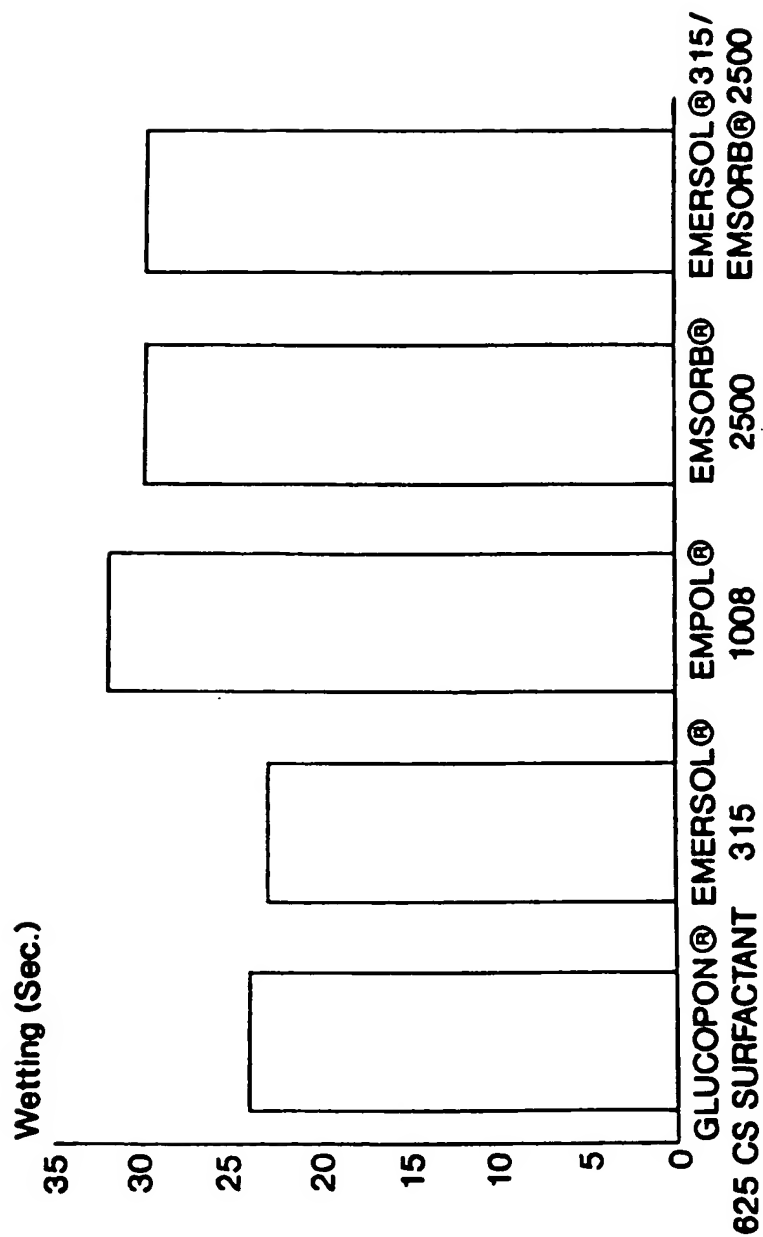


FIG. 7

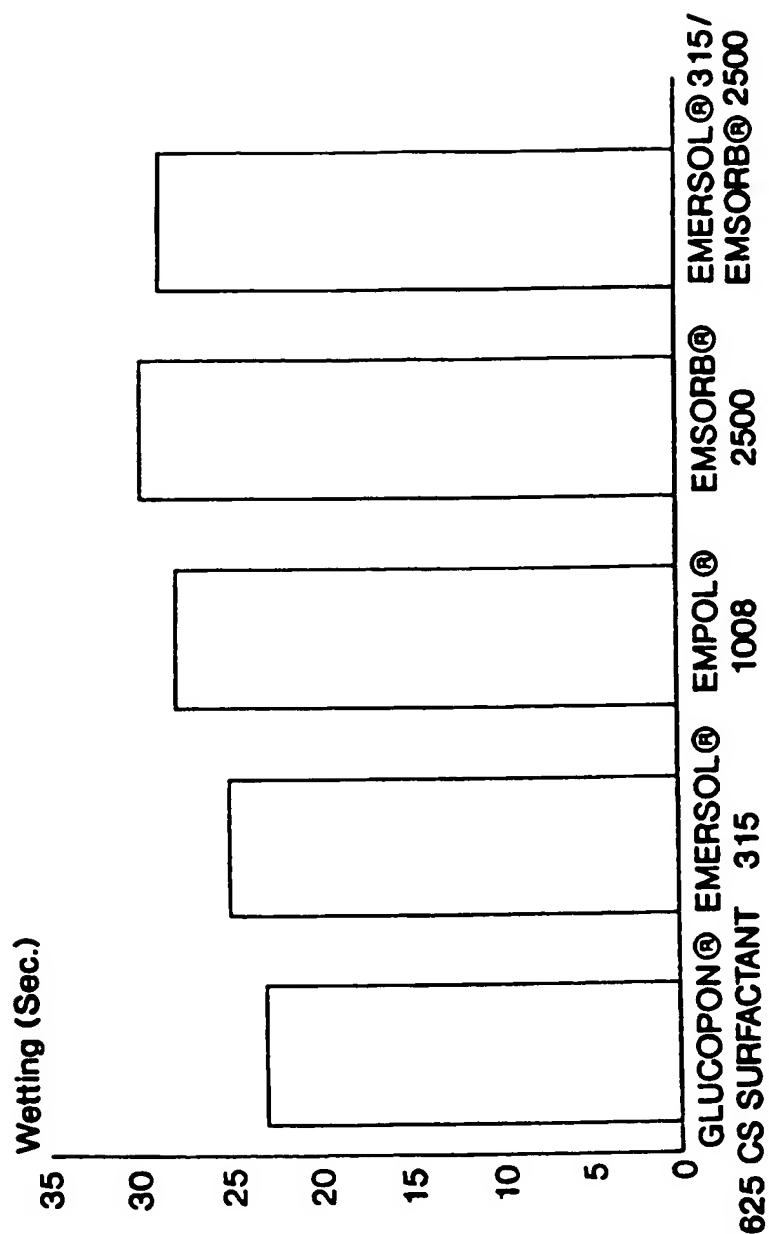


FIG. 8

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US95/13999

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C07H 15/00, 17/00

US CL : 536/4.1, 18.1

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 536/4.1, 18.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|--------------|---|------------------------|
| X -- Y | US, A, 4,396,520 (PAYNE et al.) 02 August 1983, see column 1, lines 19-67 and column 2, line 50 to column 4, line 4. | 1-27 ----- 28-69 |
| X -- Y | US, A, 4,483,787 (JONES et al.) 20 November 1984, see column 2, lines 37-53 and column 3, line 63 to column 4, line 31. | 1-27 ----- 28-69 |
| X -- Y | US, A, 4,536,317 (LLENADO et al.) 20 August 1985, see column 1, line 36 to column 2, line 14 and column 3, line 60 to column 4, line 2. | 1-27 ----- 28-69 |
| X -- Y | US, A, 4,536,318 (COOK et al.) 20 August 1985, see column 1, line to column 2, line 21. | 1-27 ----- 28-69 |

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

| | | |
|--|---|--|
| * Special categories of cited documents: | T | later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention |
| *A* document defining the general state of the art which is not considered to be part of particular relevance | X | document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone |
| *E* earlier document published on or after the international filing date | Y | document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art |
| *L* document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) | 2 | document member of the same patent family |
| *O* document referring to an oral disclosure, use, exhibition or other means | | |
| *P* document published prior to the international filing date but later than the priority date claimed | | |

Date of the actual completion of the international search

22 JANUARY 1996

Date of mailing of the international search report

21 FEB 1996

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

NATHAN M. NUTTER, Jr.

Telephone No. (703) 308-1235